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WATER-IN-OIL EMULSION COMPOSITION AND EMULSION COSMETIC MATERIAL USING THE SAME

[Yuchusuigata nyuka soseibutsu oyobi kore wo mochiita nyuka keshoryo]

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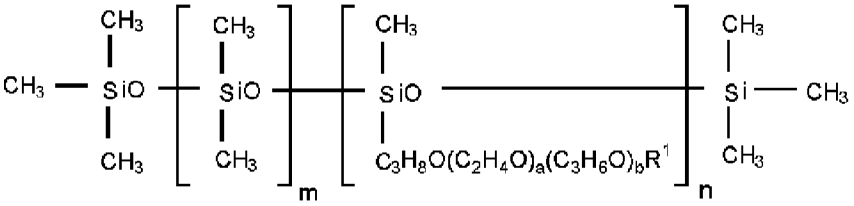
[Claims] /2

[Claim 1] A water-in-oil emulsion composition comprising (a) volatile silicone oil containing at least decamethylcyclopentanesiloxane and octamethylcyclotetrasiloxane in which the ratio of decamethylcyclopentanesiloxane to octamethylcyclotetrasiloxane is between 19:1 and 7:3 (weight ratio), (b) organically modified clay minerals, and (c) an emulsifier with an HLB of 7 or less in an oil phase (external phase).

[Claim 2] The water-in-oil emulsion composition of Claim 1, wherein the emulsifier with an HLB of 7 or less is a polyether-modified silicone surfactant expressed by Formula (I)

[Formula 1]



(I)

(where R_1 is a hydrogen atom or an alkyl group with 1-6 carbon atoms, m is a number between 1 and 150 on average, n is a number between 1 and 50 on average, and a and b are numbers between 0 and 35 on average).

[Claim 3] The water-in-oil emulsion composition of Claim 1 or Claim 2, wherein the total amount of decamethylcyclopentanesiloxane and octamethylcyclotetrasiloxane in the volatile silicone oil is 80 wt% or more.

[Claim 4] The water-in-oil emulsion composition in any one of

Claims 1 through 3, wherein the total amount of water in the composition is 50 wt% or more.

[Claim 5] An emulsion cosmetic material comprising the water-in-oil emulsion composition in any one of Claims 1 through 4.

[Detailed Description of the Invention]

[0001] [Technical Field of the Invention]

The present invention relates to a water-in-oil emulsion composition and an emulsion cosmetic material using this composition. More specifically, the present invention relates to a water-in-oil emulsion composition and an emulsion cosmetic material using this composition in which a good emulsifying state is obtained that does not change with the temperature or over time, in which the composition remains stable at temperatures lower than 0°C, and in which the feel remains fresh when used.

[0002] [Prior Art]

There have been many attempts to improve the usability and temperature stability of water-in-oil (W/O) emulsion compositions. An oil-in-water emulsion composition was disclosed in Kokai No. 61-129033 in which an organically modified clay material is used to obtain a good emulsifying state with superior usability and temperature stability even when the internal water phase ratio is high and even when the amount of solid/semi-solid oil in the oil phase has been greatly reduced. In addition, the smooth and fresh feel can be improved by using volatile silicone oil as the oil phase component.

[0003] However, because volatile silicone oil has a low melt

point, water-in-oil emulsion compositions using volatile silicone oil as the main component tend to freeze at low temperatures. Water-in-oil emulsion compositions tend to destroy the emulsion once frozen and the emulsion state cannot be recovered when thawed.

[0004] [Problem Solved by the Invention]

There is strong interest in developing a water-in-oil emulsion composition containing volatile silicone oil as the main component with good low-temperature stability, especially stability when frozen, and good feel when used.

[0005] The present inventors conducted extensive research to solve this problem. As a result, they discovered that a water-in-oil emulsion composition containing volatile silicone oil as the main component could be obtained that prevented freezing at low temperatures and that had superior feel when used if a particular amount of decamethylcyclopentanesiloxane and octamethylcyclotetrasiloxane was compounded with the volatile silicone oil. The present invention is a product of this discovery.

[0006] [Means of Solving the Problem]

In other words, the present invention is a water-in-oil emulsion composition comprising (a) volatile silicone oil containing at least decamethylcyclopentanesiloxane and octamethylcyclotetrasiloxane in which the ratio of decamethylcyclopentanesiloxane to octamethylcyclotetrasiloxane is between 19:1 and 7:3 (weight ratio), (b) organically modified clay minerals, and (c) an emulsifier with an HLB of 7 or less in an oil phase (external phase).

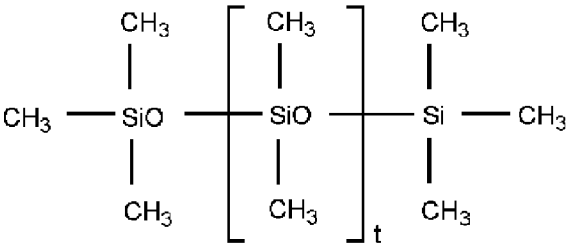
[0007] The present invention is also an emulsion cosmetic material comprising this water-in-oil emulsion composition.

[0008] [Embodiment of the Invention]

The following is a detailed explanation of the present invention.

[0009] The volatile silicone oil used as component (a) is preferably a low boiling point silicone oil with a boiling point at room temperature between 60 and 260°C. Specific examples include the silicone oils expressed by Formula (II) and Formula (III).

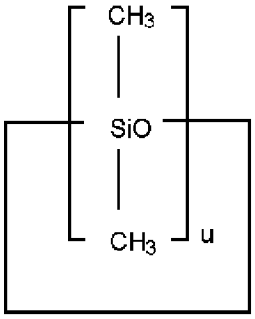
[0010] [Formula 2] /3



(II)

[0011] (Here, t is a whole number between 0 and 3.)

[0012] [Formula 3]



(III)

[0013] (Here, u is a whole number between 3 and 7.) The cyclic silicone oils expressed in Formula (III) are preferred, especially those in which u in Formula (III) is 4 through 6. Examples include decamethylcyclohexanesiloxane, octamethylcyclotetrasiloxane and decamethylcyclopentanesiloxane.

[0014] In the present invention, at least decamethylcyclopentanesiloxane [u = 5 in Formula (III)] and octamethylcyclotetrasiloxane [u = 4 in Formula (III)] have to be included in the volatile silicone oil, and the ratio of decamethylcyclopentanesiloxane to octamethylcyclotetrasiloxane in the volatile silicone oil has to be between 19:1 and 7:3 (weight ratio). When both are within this compositional ratio, a water-in-oil emulsion composition can be obtained with superior base stability, low temperature stability at 0°C or lower, and good freshness when used. If too much decamethylcyclopentanesiloxane is used, the water in the emulsion composition freezes at low temperatures and stability cannot be maintained. When too little is used, the water and the octamethylcyclotetrasiloxane freeze at low temperatures and stability cannot be maintained.

[0015] Therefore, the total amount of decamethylcyclopentanesiloxane and octamethylcyclotetrasiloxane in the volatile silicone oil is preferably 80 wt% or more.

[0016] The amount of component (a) in the emulsion composition of the present invention is preferably between 5 and 40 wt%. If less than 5 wt% is used, the volatile silicone oil does not have the desired fresh feel and the base stability is poor due to the high internal-external phase ratio. If more than 40 wt% is used, the emulsion has an oily feel, the viscosity of the base is poor due to the low internal-external phase ratio, and the base stability is poor.

[0017] There are no particular restrictions on the type of

organically modified clay mineral used as component (b). However, in the present invention, a cation modified clay mineral obtained by processing a water-swollen clay mineral treated with a quaternary ammonium salt-type cationic surfactant is suitable.

[0018] The water-swollen clay mineral can be a clay mineral such as a colloidal water-containing aluminum silicate with a three-layer structure such as a lamellar silicic acid salt smectite mineral belonging expressed by Formula (IV).

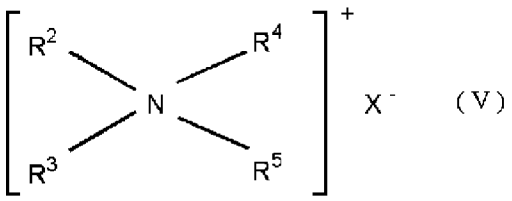
[0019] [Formula 4]

$$(X,Y)_A(Si,Al)_4O_{10}(OH)_2Z_{1/3} \cdot nH_2O \quad (IV)$$

[0020] (Here, X is Al, FeIII, MnIII or CrIII; Y is Mg, FeII, Ni, Zn or Li; Z is K, Na or Ca; and A is a number between 2 and 3). Preferred examples include natural or synthetic [where the (OH) group in Formula (IV) is substituted by F] montmorillonites such as montmorillonite, saponite and hectorite; commercial products such as Kunibia and Smectone (both from Kunichemi Kokyo Co. Ltd.), V-Gum (Vanderbilt Chemical Corp.) or Laponite (Laporte Corp.); and synthetic micas known by the name sodium silicic mica or lithium teniolite (commercial products include Diamonite and Fluor-tetrasilicic Mica (both from Topy Co. Ltd.). These water-swollen clay minerals can be used alone or in combinations of two or more.

[0021] The quaternary ammonium salt-type cationic surfactant can be a compound expressed by Formula (V).

[0022] [Formula 5] /4



(V)

[0023] (Here, R^2 is an alkyl group or benzyl group with 10 to 22 carbon atoms; R3 is a methyl group or an alkyl group with 10 to 22 carbon atoms; R4 and R5 are an alkyl groups or hydroxyalkyl group with one to three carbon atoms; and X is a halogen atom or methylsulfate residue). Specific examples include dodecy trimethyl ammonium chloride, myristyl trimethyl ammonium chloride, cetyl trimethyl ammonium chloride, stearyl trimethyl ammonium chloride, alkyl trimethyl ammonium chloride, behenyl trimethyl ammonium chloride, myristyl dimethylethyl ammonium chloride, cetyl dimethylethyl ammonium chloride, stearyl dimethylethyl ammonium chloride, alkyl dimethylethyl ammonium chloride, behenyl dimethylethyl ammonium chloride, myristyl diethylmethyl ammonium chloride, cetyl diethylmethyl ammonium chloride, stearyl diethylmethyl ammonium chloride, alkyl diethylmethyl ammonium chloride, behenyl diethylmethyl ammonium chloride, benzyl dimethyl myristyl ammonium chloride, benzyl dimethyl cetyl ammonium chloride, benzyl dimethyl stearyl ammonium chloride, benzyl dimethyl behenyl ammonium chloride, benzyl methylethyl cetyl ammonium chloride, benzyl methylethyl stearyl ammonium chloride, distearyl dimethyl ammonium chloride, dibehenyl dihydroxyethyl ammonium chloride, bromides of these compounds, and dipalmitoyl propylethyl ammonium methyl sulfide. These quaternary ammonium salt-type cationic

surfactants can be used alone or in combinations of two or more.

[0024] Quaternary ammonium salt-type cation-modified clay minerals are available commercially. Examples include Bentone 38 (National Red Co. Ltd.) and V-Gum Ultra (Vanderbilt Chemical Corp.).

[0025] When a water-swollen clay mineral is treated with a quaternary ammonium salt-type cationic surfactant, a non-ionic surfactant can also be used in the treatment.

[0026] Examples of non-ionic surfactants include ethylene-oxide adduct-type surfactants, polyhydric alcohol fatty acid ester-type surfactants, and non-ionically modified silicone surfactants.

[0027] Examples of ethylene-oxide adduct-type surfactants include ether-type surfactants such as polyoxyethylene 2-30 mole adduct [POE (2-30)] oleyl ether, POE (2-35) stearyl ether, POE (2-20) lauryl ether, POE (1-20) alkylphenyl ether, POE (6-18) behenyl ether, POE (5-25) 2-decylpentadecyl ether, POE (3-20) 2-decyltetradecyl ether, POE (3-20) 2-decyltetradecyl ether, and POE (8-16) 2-octyldecyl ethyl; ester-type surfactants such as POE (4-60) hardened castor oil, POE (3-14) fatty acid monoester, POE (6-30) fatty acid diester, and POE (5-20) sorbitan fatty acid ester; and ether ester-type surfactants such as POE (2-30) glyceryl monoisostearate, POE (10-60) glyceryl triisostearate, POE (7-50) hardened castor oil monoisostearate, POE (12-60) hardened castor oil triisostearate.

[0028] Specific examples of polyhydric alcohol fatty acid ester-type surfactants include polyglycerin fatty acid esters such as decaglyceryl tetraoleate, hexaglyceryl triisostearate, tetraglyceryl

diisostearate, diglyceryl diisostearate; and glyceryl monoisostearate and glyceryl monooleate.

[0029] Specific examples of non-ionically modified silicone surfactants include modified silicones such as dimethylpolysiloxane-polyoxyalkylene copolymers.

[0030] Among these ethylene-oxide adduct-type surfactants, the following are preferred: polyglycerin fatty acid esters above triglycerins such as decaglyceryl tetraoleate, hexaglyceryl triisostearate and tetraglyceryl diisostearate; POE adduct ether-type surfactants such as POE (2-12) oleyl ether, POE (3-12) stearyl ether, POE (2-10) lauryl ether, POE (2-10) nonylphenyl ether, POE (6-15) behenyl ether, POE (5-20) 2-decylpentadecyl ether, POE (5-17) 2-decyltetradecyl ether, and POE (8-16) 2-octyldecyl ether; POE adduct ester-type surfactants such as POE (10-20) hardened castor oil, POE (5-14) oleic acid monoester, POE (6-20) oleic acid diester, and POE (5-10) sorbitan oleic acid ester; and POE adduct ether ester-type surfactants such as POE (3-15) glyceryl monoisostearate and POE (10-40) glyceryl triisostearate. These ethylene-oxide adduct-type surfactants can be used alone or in combinations of two or more. /5

[0031] The organically modified clay minerals used in the present invention can be dispersed in a low boiling point solvent such as water, acetone or a lower alcohol and stirred with a quaternary ammonium salt-type cationic surfactant to remove the low boiling point solvents. A water-swollen clay mineral can also be compounded with a quaternary cationic surfactant to modify it organically during the

treatment. Clay minerals obtained in this manner are preferred. If desired, a non-ionic surfactant can be combined with the quaternary ammonium-salt cationic surfactant.

[0032] The amount of quaternary ammonium salt-type cationic surfactant used should be 60 to 140 milligram equivalents (meq) per 100 g of water-swollen clay mineral. The amount of quaternary non-ionic surfactant used should be 5 to 100 g, preferably 15 to 50 g, per 100 g of water-swollen clay mineral.

[0033] The amount of component (b) compounded should be 0.1 to 10 wt%, and preferably 0.1 to 5 wt%, of the emulsion composition of the present invention. If less than 0.1 wt%, the organically modified clay material has no effect and a stable emulsion composition cannot be obtained. If more than 10 wt% is used, the viscosity of the prepared emulsion is high, the extension is poor, and practical problems such as roughness and a lack of transparency occur. These organically modified clay materials can be used alone or in combinations of two or more.

[0034] The emulsifier used as component (c) should have an HLB of 7 or less. If the HLB exceeds 7, the emulsifier is too hydrophilic and a stable water-in-oil emulsion composition cannot be obtained.

[0035] In the present invention, HLB is calculated using the Kawakami method expressed by the following equation.

[0036] [Equation 1]

$$HLB = 7 + 11.7 \cdot \log (MW/MO)$$

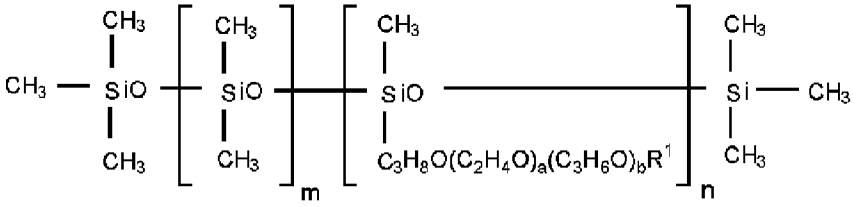
[0037] (Here, MW is the molecular weight of the hydrophilic

groups and MO is the molecular weight of the lipophilic groups.

[0038] Emulsifiers with an HLB of 7 or less include sorbitan fatty acid esters such as sorbitan monolaurate, sorbitan monoisostearate and sorbitan tristearate; glycerin fatty acid esters such as glycerol monostearate and glycerol monooleate; polyoxyethylene hardened castor oils such as POE (5) hardened castor oil, POE (7.5) hardened castor oil and POE (10) hardened castor oil; and polyether-modified silicone surfactants.

[0039] Use of a polyether-modified silicone surfactant expressed by Formula (I) is preferred.

[0040] [Formula 6]



(I)

[0041] (Here, R^1 is a hydrogen atom or an alkyl group with 1-6 carbon atoms, m is a number between 1 and 150 on average, n is a number between 1 and 50 on average, and a and b are numbers between 0 and 35 on average). Examples of commercially available polyether-modified silicone surfactants include Silicone SC1014M (Shinetsu Chemical Co. Ltd.) and Toray Silicone SH-3771 (Toray Dow Corning Co. Ltd.).

[0042] The amount of component (c) compounded should be 0.1 to 15 wt%, and preferably 0.5 to 10 wt%, of the emulsion composition of the present invention. If less is compounded, a stable emulsion cannot be

obtained. If more is used, practical problems such as stickiness and a lack of freshness occur. One or more component can be used as component (c).

[0043] Component (c) should be added to the composition of the present invention adsorbed into the organically modified clay mineral serving as component (b). In other words, when the water-in-oil emulsion composition or emulsion cosmetic material of the present invention is manufactured, the organically modified clay mineral can be treated with the emulsifier with an HLB of 7 or less serving as component (c) in order to compound component (c) such that the emulsifier with an HLB of 7 or less is adsorbed into the organically modified clay mineral.

[0044] In addition to the necessary components, other components can be added to the external phase (oil phase) in an amount that does not compromise the effect of the present invention. These include components commonly used in cosmetics and medicines such as oily components, oily polymers, powders and macromolecular granules.

[0045] In addition to water, other components can be added to the internal phase (water phase) in an amount that does not compromise the effect of the present invention. These include components commonly used in cosmetics and medicines: water-soluble materials such as Vitamin B, Vitamin C, vitamin derivatives, pantothenic acid and derivatives, and biotin; buffering agents such as sodium glutamate, arginine, aspartic acid, citric acid, tartaric acid and lactic acid; chelates such as EDTA; water-soluble UV absorbers; and various types

of pigments. There are no particular restrictions. /6

[0046] From the standpoint of usability, the total amount of water used in the composition including both the water phase and the oil phase should be 50 wt% or more of the entire composition.

[0047] The moisturizing effect of the water-in-oil emulsion composition of the present invention can be increased by including a polyhydric alcohol, polyhydric alcohol derivative or moisturizer in a compositional amount that does not compromise the effect of the present invention.

[0048] Examples of polyhydric alcohols and derivatives include dihydric alcohols such as ethylene glycol, propylene glycol, trimethylene glycol, 1,2-butylene glycol, 1,3-butylene glycol, tetramethylene glycol, 2,3-butylene glycol, pentamethylene glycol, 2-butene-1,4-diol, hexylene glycol, and octylene glycol; trihydric alcohols such as glycerin, trimethyol propane and 1,2,6-hexanetriol; tetrahydric alcohols such as pentaerythritol; pentahydric alcohols such as xylitol; hexahydric alcohols such as sorbitol and mannitol; polyhydric alcohol copolymers such as diethylene glycol, dipropylene glycol, triethylene glycol, polypropylene glycol, triglycerin, tetraglycerin, and polyglycerin; dihydric alcohol alkyl ethers such as ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, ethylene glycol monobutyl ether, ethylene glycol monophenyl ether, ethylene glycol monohexyl ether, ethylene glycol mono-2-methylhexyl ether, ethylene glycol isoamyl ether, ethylene glycol benzyl ether, ethylene glycol isopropyl ether, ethylene glycol dimethyl ether,

ethylene glycol diethyl ether, and ethylene glycol dibutyl ether; dihydric alcohol alkyl ethers such as diethylene glycol monomethyl ether, diethylene glycol monoethyl ether, diethylene glycol monobutyl ether, diethylene glycol dimethyl ether, diethylene glycol diethyl ether, diethylene glycol dibutyl ether, diethylene glycol methylethyl ether, triethylene glycol monomethyl ether, triethylene glycol monoethylether, triethylene glycol monoethylether, propylene glycol monomethyl ether, propylene glycol monoethylether, propylene glycol monobutyl ether, propylene glycol monoisopropyl ether, dipropylene glycol methyl ether, dipropylene glycol ethylether, and dipropylene glycol butyl ether; dihydric alcohol ether esters such as ethylene glycol monomethyl ether acetate, ethylene glycol monoethyl ether acetate, ethylene glycol monobutyl ether acetate, ethylene glycol monophenyl ether acetate, ethylene glycol diadipate, ethylene glycol disuccinate, ethylene glycol monoethyl ether acetate, diethylene glycol monobutyl ether acetate, propylene glycol monomethyl ether acetate, propylene glycol monoethyl ether acetate, propylene glycol monopropyl ether acetate, and propylene glycol monophenyl ether acetate; glycerin monoalkyl ethers such as xylyl alcohol, selachyl alcohol and batyl alcohol; and sugar alcohols such as sorbitol, multitol, maltotriose, mannitol, sucrose, erythritol, glucose, galactose, amylolytic sugars, maltose, xylitose, and amylolytic reduced alcohols; and other such as glysolids, tetrahydrofurfuryl alcohol, POE tetrahydrofurfutyl alcohol, POP butylether, POP·POE butylether, chilled polyoxypropylene glycol ether, POP glycerin ether,

POP glycerin ether phosphate, and POP·POE pentaerythritol ether.

[0049] Examples of moisturizers include chondroitin sulfuric acid, hyaluronic acid, mucoitinsulfuric acid, charonin sulfuric acid, atelocollagen, cholesteryl-12-hydroxystearate, sodium lactate, bile acid mono salts, dl-pyrrolidone carboxylic acid mono salts, short-chain soluble collagens, Common Yarrow extract and Achillea millefolium extract.

[0050] A water-soluble polymer can be compounded in a compositional range that does not compromise the usability of the emulsion composition of the present invention. These water-soluble polymers can be natural water-soluble polymers, semi-synthetic water-soluble polymers and inorganic water-soluble polymers.

[0051] Examples of natural water-soluble polymers include plant-based water-soluble polymers such as gum arabic, tragacanth gum, galactan, guar gum, carob gum, karaya gum, carageenan, pectin, agar-agar, quince seed (marmelo), algae colloid (brown algae extract), starch (rice, maize, potato, wheat) and glycyrrhizinic acid; microorganism-based water-soluble polymers such as xanthan gum, dextran, succinoglycan and pullulan; and animal-based water-soluble polymers such as collagen, cassein, albumin and gelatin.

[0052] Semi-synthetic water-soluble polymers include starch-based water-soluble polymers such as carboxymethyl starch and /7
methylhydroxypropyl starch; cellulose-based water-soluble polymers such as methyl cellulose, nitrocellulose, ethyl cellulose, methylhydroxypropyl cellulose, hydroxyethyl cellulose, cellulose

sodium sulfate, hydroxypropyl cellulose, sodium carboxymethyl cellulose (CMC), crystal cellulose and cellulose powder; and alginic acid-based water-soluble polymers such as sodium alginate and alginic acid propylene glycose ester.

[0053] Synthetic water-soluble polymers include vinyl-based water-soluble polymers such as polyvinyl alcohol, polyvinyl methyl ether, polyvinyl pyrrolidone and carboxyvinyl polymers (commercial name: Carbopol); polyoxyethylene-based water-soluble polymers such as polyethylene glycol (mol. wt. 20,000, 4,000, 6,000); polyoxyethylene-polyoxypropylene copolymer-based water-soluble polymers; acrylic-based water-soluble polymers such as sodium polyacrylate, polyethyl acrylate and polyacrylamide; polyethyleneimines; and cationic polymers.

[0054] Inorganic water-soluble polymers include bentonite, magnesium aluminate silicate (commercial name: V-Gum), Laponite, hectolite and anhydrous silicic acid.

[0055] Various types of component commonly used in the fields of cosmetics and medicine can also be included in the emulsion composition of the present invention.

[0056] An anionic surfactant, non-ionic surfactant, cationic surfactant or amphoteric surfactant can be used in a compositional range that does not compromise the effect of the present invention.

[0057] The water-in-oil emulsion composition of the present invention can be used in any product in the fields of cosmetics and medicines such as skin cosmetics, hair cosmetics and topical medicines. Because of its usability, a preferred use is as an emulsion

cosmetic material.

[0058] [Working Examples]

The following is a more detailed explanation of the present invention with reference to working examples. The present invention is by no means restricted to these working examples. All parts are weight percentages.

[0059] First, the evaluation methods used on the working examples will be explained.

[0060] [Usability (Moistness)] A panel of ten women used the emulsion compositions (samples) in the working examples and comparative examples and evaluated the compositions according to the following evaluation scale.

(Evaluation)

◎: 8 out of 10 or more found the composition to be moist

○: 6 out of 10 or more found the composition to be moist

Δ: 4 out of 10 or more found the composition to be moist

×: 3 out of 10 or less found the composition to be moist

[0061] [Usability (Freshness)] A panel of ten women used the emulsion compositions (samples) in the working examples and comparative examples and evaluated the compositions according to the following evaluation scale.

(Evaluation)

◎: 8 out of 10 or more found the composition to be fresh

○: 6 out of 10 or more found the composition to be fresh

Δ: 4 out of 10 or more found the composition to be fresh

×: 3 out of 10 or less found the composition to be fresh

[0062] [Stability] The samples obtained in the working examples and comparative examples were stored at -10°C, 0°C, room temperature, 37°C and 50°C for a month. The emulsion state was then visually inspected and evaluated according to the following standard.

(Evaluation)

◎: Normal

○: Some Oil Floating

Δ: Oil Floating

×: Separated

[0063] (Working Examples 1-4, Comparative Examples 1-4) The emulsion compositions (creams) shown in Tables 1-2 were prepared in the usual manner. The usability (moistness, freshness) and the stability of the compositions were then evaluated using the evaluation methods described above. The results are shown in Table 3.

[0064] [Table 1]

	A 成　　分	B 実施例１	実施例２	実施例３	実施例４
C	デカメチルシクロペンタシロキサン	14.0	17.0	27.0	21.0
D	オクタメチルシクロテトラシロキサン	5.0	2.0	3.0	9.0
E	ドデカメチルシクロヘキサシロキサン	1.0	1.0	－	－
F	ポリエーテル変性シリコーン系界面活性剤 (*)	3.0	3.0	3.0	3.0
G	有機変性粘土鉱物 (**)	2.0	2.0	1.5	1.5
H	精製水	K 残 部	残 部	残 部	残 部
I	ジプロピレングリコール	5.0	5.0	5.0	5.0
J	Ｌ－グルタミン酸ソーダ	0.5	0.5	0.5	0.5

Key: A = Component, B = Working Example, C = Decamethylcyclopentanesiloxane, D = Octamethylcyclotetrasiloxane, E = Decamethylcyclohexanesiloxane, F = Polyethyer-Modified Silicone Surfactant (*), G = Organically Modified Clay Mineral (**), H = Purified Water, I = Dipropylene Glycol, J = L-Glutamic Acid Soda, K = Balance

[0065] [Table 2] /8

A 成　分	B 比較例1	比較例2	比較例3	比較例4
C デカメチルシクロペンタシロキサン	15.0	15.0	30.0	3.0
D オクタメチルシクロテトラシロキサン	10.0	0.1	15.0	0.1
E ドデカメチルシクロヘキサシロキサン	—	—	5.0	—
F ポリエーテル変性シリコーン系界面活性剤(*)	3.0	3.0	4.0	1.0
G 有機変性粘土鉱物(**)	2.0	2.0	2.0	1.0
H 精製水	K 残 部	残 部	残 部	残 部
I ジプロピレングリコール	5.0	5.0	5.0	5.0
J L－グルタミン酸ソーダ	0.5	0.5	0.5	0.5

Key: A = Component, B = Working Example, C = Decamethylcyclopentanesiloxane, D = Octamethylcyclotetrasiloxane, E = Decamethylcyclohexanesiloxane, F = Polyethyer-Modified Silicone Surfactant (*), G = Organically Modified Clay Mineral (**), H = Purified Water, I = Dipropylene Glycol, J = L-Glutamic Acid Soda, K = Balance

[0066] In Table 1 and Table 2, the polyether-modified silicone surfactant (*) is Silicone SC9450N (Shinetsu Chemical Industry Co. Ltd.) and the organically modified clay mineral (**) is Bentone 38 (National Red Co. Ltd.).

[0067] [Table 3]

A 評価		B 実施例				C 比較例			
		1	2	3	4	1	2	3	4
使用性 D	みずみずしさ F	◎	◎	○	○	△	◎	×	◎
	さっぱり感 G	◎	◎	○	○	○	◎	△	◎
安定性 E	−10℃	◎	◎	◎	◎	I 凍結	凍結	凍結	凍結
	0℃	◎	◎	◎	◎	凍結	◎	◎	○
	室温 H	◎	◎	◎	◎	◎	◎	○	○
	37℃	◎	◎	◎	◎	◎	◎	△	×
	50℃	◎	◎	◎	◎	◎	◎	×	×

Key: A = Evaluation, B = Working Example, C = Comparative Example, D = Usability, E = Stability, F = Moist, G = Fresh, H = Room Temperature, I = Frozen

[0068] From the results in Table 3, the products of the present invention had superior usability (moist, fresh) and superior temperature stability.

[0069] [Preparation] (6) through (10) and (12) were mixed together at room temperature and evenly dispersed in the oil phase. (2), (4) and (5) were added to (1), and (11) dissolved in (3) was added to obtain the water phase. The water phase was gradually added to the oil phase and uniformly dispersed in a homomixer to obtain emulsified particles. The resulting water-in-oil emulsion cosmetic material was made into a moisturizing cream.

(Working Example 5: Moisturizing Cream) (wt%)

(1) Purified Water	Balance
(2) Glycerin	1.0
(3) 1,3-Butyleneglycol	5.0
(4) Trehalose	3.0
(5) Dynamite Glycerin	5.0
(6) Decamethylcyclopentanesiloxane	16.0
(7) Octamethylcyclotetrasiloxane	4.0
(8) Polyether-Modified Silicone Surfactant (Silicone SC9450N from Shinetsu Chem. Co. Ltd.)	2.0
(9) Organically Modified Clay Mineral (Bentone 27 [sic] from National Red Co. Ltd.)	3.0
(10) Vitamin E-Acetate	1.0
(11) Methylparaben	Suitable Amount
(12) Fragrance	Suitable Amount

[0070] When the usability and stability of this moisturizing cream was evaluated, both the usability and stability were stable.

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(Working Example 6: O/W/O Cream) (wt%)

(1) Polyoxyethylene Hardened Castor Oil	2.0
(2) Behenyl Alcohol	6.0
(3) Liquid Paraffin	12.0
(4) Vaseline	3.0
(5) Vitamin E-Acetate	1.0
(6) Ion-Exchange Water	Balance
(7) 1,3-Butyleneglycol	5.0
(8) Ascorbic Acid	0.5
(9) Arbutin	2.0
(10) Phenoxyethanol	Suitable Amount
(11) Polyether-Modified Silicone Surfactant (Silicone SC9450N from Shinetsu Chem. Co. Ltd.)	2.0
(12) Organically Modified Clay Mineral (Bentone 38 from National Red Co. Ltd.)	3.0
(13) Decamethylcyclopentanesiloxane	8.0
(14) Octamethylcyclotetrasiloxane	3.0
(15) Fragrance	Suitable Amount

[Preparation] An O/W emulsion was obtained with (1) through (10). This O/W emulsion was gradually added to an oil phase in which (11)

through (15) were uniformly dispersed, and the phases were uniformly dispersed using a disperser to obtain a stable O/W/O emulsion. This emulsion had good stability and superior usability.

[0072] [Effect of the Invention]

As explained above, the present invention provides a water-in-oil emulsion composition and emulsion cosmetic material with good stability, especially low-temperature stability, as well as good usability such as moistness and freshness.